

इंटरनेट

मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

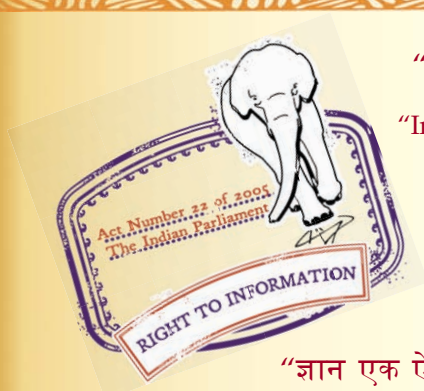
“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

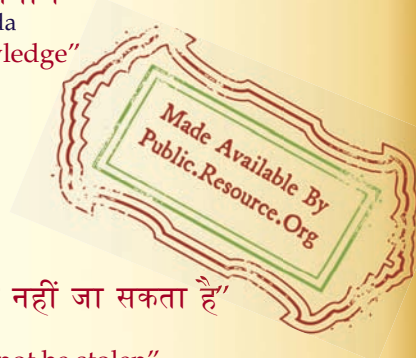
IS 4646-1 (1968): Methods of chemical analysis of copper phosphorus brazing alloys, Part 1 Analysis for silver and copper [MTD 8: Copper and Copper Alloys]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

BLANK PAGE



Indian Standard

METHODS OF CHEMICAL ANALYSIS OF COPPER-PHOSPHORUS BRAZING ALLOYS

PART I ANALYSIS FOR SILVER AND COPPER

(Second Reprint JULY 1996)

UDC 621.791.36:669.35'779'22:543

© Copyright 1968

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF COPPER-PHOSPHORUS BRAZING ALLOYS

PART I ANALYSIS FOR SILVER AND COPPER

Methods of Chemical Analysis Sectional Committee, SMDC 2

Chairman

DR R. K. DUTTA

Representing

Durgapur Steel Plant, Durgapur

Members

- | | |
|------------------------------------------------------------------------|-------------------------------------------------------------|
| SHRI K. L. BANERJEE | National Test House, Calcutta |
| SHRI N. C. SEN GUPTA (<i>Alternate</i>) | |
| SHRI J. BHATTACHARJEE | Ministry of Defence (DGI) |
| DR H. P. BHATTACHARYA | National Metallurgical Laboratory (CSIR), Jamshedpur |
| SHRI A. C. BHOWMIK | Durgapur Steel Plant, Durgapur |
| DR U. N. BHRANY | Indian Iron & Steel Co Ltd, Burnpur |
| SHRI A. C. MUKHERJEE (<i>Alternate</i>) | |
| DR B. N. CHAKRAVARTHI | Bhilai Steel Plant, Bhilai |
| DR V. N. RAO (<i>Alternate</i>) | |
| SHRI A. K. CHATTERJEE | The Binani Metal Works Limited, Calcutta |
| CHEMIST & METALLURGIST, SOUTH CENTRAL RAILWAY, SECUNDERABAD | Ministry of Railways |
| ASSISTANT DIRECTOR (MET), RDSO, CHITTARANJAN (<i>Alternate</i>) | |
| DR A. N. CHOWDHURY | Geological Survey of India, Calcutta |
| SHRI B. N. BHATTACHARYA (<i>Alternate</i>) | |
| SHRI S. N. DASGUPTA | Directorate General of Supplies and Disposals, New Delhi |
| SHRI K. L. MURTY (<i>Alternate</i>) | |
| DIRECTOR OF SCIENTIFIC RE- SEARCH (NAVY) | Naval Headquarters |
| SHRI C. R. GOIL | Ferro Alloys Corporation Ltd, Shreeramnagar |
| SHRI G. S. GOURISHANKAR | Directorate General of Ordnance Factories, Calcutta |
| SHRI J. M. KAWATHEKAR (<i>Alternate</i>) | |
| SHRI S. S. HONAVAR | Italab Private Ltd, Bombay |
| SHRI J. C. DEY (<i>Alternate</i>) | |
| SHRI J. P. PATEL (<i>Alternate</i>) | |
| DR N. JAYARAMAN | Essen & Co, Bangalore |
| SHRI M. R. G. SHARMA (<i>Alternate</i>) | |

(Continued on page 2)

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

IS : 4646 (Part I) - 1968

(Continued from page 1)

Members

DR M. K. JOSHI

SHRI S. L. N. ACHARYULU (Alternate)

DR S. KUMAR

SHRI B. K. AGARWAL (Alternate)

SHRI N. C. MITRA

SHRI M. M. MOUDGILL

SHRI N. MAZUMDAR (Alternate)

SHRI K. P. ROY CHOWDHURY

SHRI A. SANGAMESWARA RAO

DR P. R. SUBBARAMAN

DR V. S. PANSARE (Alternate)

DR CH. VENKATESWARLU

SHRI M. R. VERMA

SHRI R. K. SRIVASTAVA,
Head (Struc & Met)

Representing

Defence Metallurgical Research Laboratory,
Hyderabad

Central Glass and Ceramic Research Institute
(CSIR), Calcutta

India Government Mint (Ministry of Finance),
Calcutta

Indian Non-Ferrous Metals Manufacturers'
Association, Calcutta

The Eyre Smelting Private Ltd, Calcutta

Indian Metals and Ferro Alloys Ltd, Koraput

National Chemical Laboratory (CSIR), Poona

Bhabha Atomic Research Centre, Bombay

National Physical Laboratory (CSIR), New
Delhi

Director General, ISI (Ex-officio Member)

Secretary

SHRI S. L. BALI

Deputy Director (Met), ISI

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF COPPER-PHOSPHORUS BRAZING ALLOYS

PART I ANALYSIS FOR SILVER AND COPPER

0. FOREWORD

0.1 This Indian Standard (Part I) was adopted by the Indian Standards Institution on 14 June 1968, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 For the correct analysis of copper-phosphorus brazing alloys, which are mainly used for joining copper and copper alloys, certain methods have been laid down in this standard. In this part, the methods for analysis of silver and copper have been prescribed. The method for the determination of phosphorus is being investigated and will be covered in Part II of this standard.

0.3 In the formulation of this standard, due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by deriving assistance from the '1967 Book of ASTM Methods for chemical analysis of metals: Part 32' issued by the American Society for Testing and Materials.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part I) prescribes methods for chemical analysis of silver and copper in various grades of copper-phosphorus brazing alloys as specified in IS : 2927-1964†.

2. SAMPLING

2.1 Laboratory Sample — It shall be drawn and prepared in accordance with IS : 1817-1961‡.

*Rules for rounding off numerical values (revised).

†Specification for brazing alloys.

‡Methods of sampling non-ferrous metals for chemical analysis.

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. DETERMINATION OF SILVER BY THE GRAVIMETRIC METHOD

4.1 **Outline of the Method** — The sample is dissolved in nitric acid and silver precipitated as silver chloride by means of hydrochloric acid. The precipitate is filtered, washed, dried and weighed.

4.2 Reagents

4.2.1 *Dilute Nitric Acid* — 1 : 1 and 3 : 97 (*v/v*).

4.2.2 *Dilute Hydrochloric Acid* — 1 : 9 (*v/v*).

4.3 Procedure

4.3.1 Transfer 2 g of an accurately weighed sample into a 250-ml beaker. Add 25 ml of dilute nitric acid (1 : 1). Heat gently to dissolve and boil to remove the brown fumes. Cool to room temperature and dilute to 50 ml.

4.3.2 Add 25 ml of dilute hydrochloric acid (1 : 9) slowly with constant stirring. Let it stand for about an hour.

4.3.3 Decant the solution through a weighed sintered glass crucible. Wash the precipitate with warm dilute nitric acid (3 : 97) till free from chloride and decant through the crucible. Transfer the filtrate and washings to a 400-ml beaker and reserve for the determination of copper.

4.3.4 Dry the crucible and precipitate at about 110°C. Cool in a desiccator to room temperature and reweigh.

4.4 Calculation

$$\text{Silver, percent} = \frac{A \times 75.26}{B}$$

where

A = weight in g of silver chloride, and

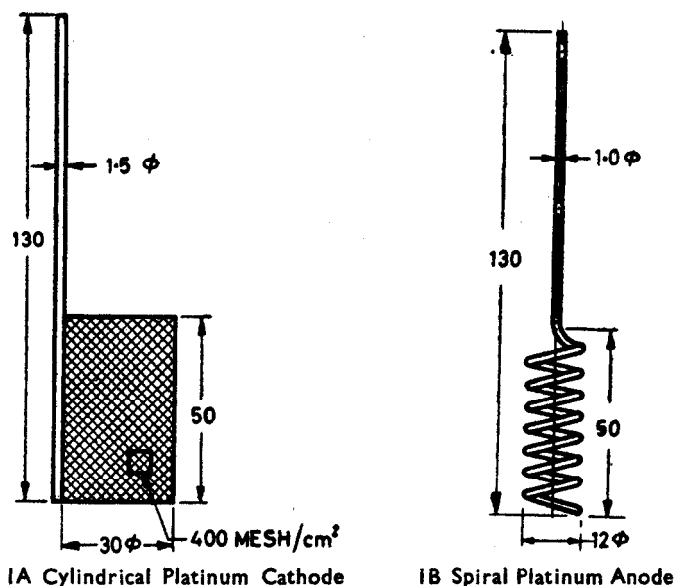
B = weight in g of the sample taken.

*Specification for water, distilled quality (*revised*).

5. DETERMINATION OF COPPER BY THE ELECTROLYTIC METHOD

5.1 Outline of the Method — After removal of silver, copper in the filtrate is deposited electrolytically and weighed.

5.2 Apparatus — The following platinum electrodes (*see* Fig. 1) are recommended but strict adherence to the shape and size of the electrodes is not essential. In order to decrease the time of deposition, one of the types of rotating forms of electrodes generally available for agitation of electrolyte may be employed.



All dimensions in millimetres.

FIG. 1 CYLINDRICAL PLATINUM CATHODE AND SPIRAL PLATINUM ANODE

5.2.1 Cathode — It may be formed either from plain or perforated sheet or from wire gauze.

5.2.1.1 Gauze cathodes, made preferably from gauze containing 400 mesh/cm², should be used. The wire used for making gauze should be approximately 0.20 mm in diameter. Cathodes should be stiffened by doubling the gauze for about 3 mm on the top and bottom or by reinforcing the gauze at the top and bottom with platinum ring or band.

5.2.1.2 The diameter and height of the cylinder should be approximately 30 and 50 mm respectively. The stem should be made from platinum alloy wire, such as platinum-iridium, platinum-rhodium or platinum-ruthenium having diameter approximately 1.5 mm. It should be flattened and welded to the entire height of the gauze. The overall height of the cathode including the stem should be approximately 130 mm.

5.2.2 Anode — Either a spiral or a gauze anode should be used. The spiral anode should be made from 1.0-mm or larger platinum wire formed into a spiral of seven coils with a height approximately 50 mm and diameter 12 mm, the overall height including the stem being 130 mm. The gauze anode should be made of the same material and of the same general design as platinum gauze cathode specified under **5.2.1**.

5.3 Reagents

5.3.1 Concentrated Sulphuric Acid — sp gr 1.84 (conforming to IS : 266-1961*).

5.3.2 Concentrated Nitric Acid — sp gr 1.42 (conforming to IS : 264-1950†).

5.3.3 Urea — solid.

5.3.4 Ethanol or Methanol — 95 percent (v/v).

5.4 Procedure

5.4.1 To the filtrate and washings reserved under **4.3.3**, add 5 ml of concentrated sulphuric acid and evaporate to fumes. Cool, dilute to 250 ml and add 10 ml of concentrated nitric acid.

5.4.2 Weigh the cathode, adjust the electrodes in the solution, and cover with a pair of split watch-glasses. Add 2 g of urea. Electrolyze at a current density of 0.6 A/dm² (at this current density, the electrolysis is conveniently carried on overnight), or at a current density of 4 A/dm² for a short period (about 2.5 hours). In the latter case, one of the types of rotating forms of electrodes generally available may be used. When the solution becomes colourless, reduce the current density to 0.3 A/dm² and continue electrolysis until the deposition of copper is complete as indicated by failure of copper to plate on the newly exposed cathode surface when the solution level is raised.

*Specification for sulphuric acid (revised).

†Specification for nitric acid.

5.4.3 Without interrupting the current, lower the beaker slowly while rinsing the electrodes with water and collecting the washings in the electrolyte. Remove the cathode quickly, rinse it with water in a beaker and then dip it in two successive baths of ethanol or methanol. Dry the cathode in an air oven at 110°C for three to five minutes, cool and reweigh the cathode. The difference in weight gives the weight of copper deposited.

5.5 Calculation

$$\text{Copper, percent} = \frac{A}{B} \times 100$$

where

A = weight in g of copper deposited, and

B = weight in g of the sample taken.

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones: 323 0131, 323 8375, 323 9402

Fax : 91 11 3234062, 91 11 3239399

Telegrams : Manaksanstha
(Common to all Offices)
Telephone

Central Laboratory :

Plot No. 20/9, Site IV, Sahibabad Industrial Area, Sahibabad 201010

8-77 00 32

Regional Offices:

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002 323 76 17

*Eastern : 1/14 CIT Scheme VII M, V.I.P. Road, Maniktila, CALCUTTA 700054 337 86 62

Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022 60 38 43

Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113 235 23 15

†Western : Manakalaya, E9, Behind Marol Telephone Exchange, Andheri (East),
MUMBAI 400093 832 92 95

Branch Offices:

'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMEDABAD 380001 550 13 48

‡Peenya Industrial Area, 1st Stage, Bangalore-Tumkur Road,
BANGALORE 560058 839 49 55

Gangotri Complex, 5th Floor, Bhadbhada Road, T.T. Nagar, BHOPAL 462003 55 40 21

Plot No. 62-63, Unit VI, Ganga Nagar, BHUBANESHWAR 751001 40 36 27

Kalaikathir Buildings, 670 Avinashi Road, COIMBATORE 641037 21 01 41

Plot No. 43, Sector 16 A, Mathura Road, FARIDABAD 121001 8-28 88 01

Savitri Complex, 116 G.T. Road, GHAZIABAD 201001 8-71 19 96

53/5 Ward No.29, R.G. Barua Road, 5th By-lane, GUWAHATI 781003 54 11 37

5-8-56C, L.N. Gupta Marg, Nampally Station Road, HYDERABAD 500001 20 10 83

E-52, Chitaranjan Marg, C-Scheme, JAIPUR 302001 37 29 25

117/418 B, Sarvodaya Nagar, KANPUR 208005 21 68 76

Seth Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road,
LUCKNOW 226001 23 89 23

Patliputra Industrial Estate, PATNA 800013 26 23 05

T.C. No. 14/1421, University P. O. Palayam, THIRUVANANTHAPURAM 695034 6 21 17

Inspection Offices (With Sale Point) :

Pushpanjali, 1st Floor, 205-A, West High Court Road, Shankar Nagar Square,
NAGPUR 440010 52 51 71

Institution of Engineers (India) Building 1332 Shivaji Nagar, PUNE 411005 32 36 35

*Sales Office is at 5 Chowringhee Approach, P.O. Princep Street,
CALCUTTA 700072 27 10 85

†Sales Office is at Novelty Chambers, Grant Road, MUMBAI 400007 309 65 28

‡Sales Office is at 'F' Block, Unity Building, Narashimaraja Square,
BANGALORE 560002 222 39 71